

**ATTACHMENT L**  
**TO**  
**BASIC ORDERING AGREEMENT**  
**ATTACHMENT 1**  
**STATEMENT OF WORK**

**GENERAL BIOASSAY**  
**SERVICES**

**Revision 0**

Reviewed For Classification

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**GENERAL BIOASSAY SERVICES**  
**Attachment L**

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## **BIOASSAY SERVICES**

### **PURPOSE**

**The purpose of this Attachment to the Basic Ordering Agreement is to provide a general standardized set of radioanalytical requirements that will ensure that high quality, consistent/comparable, and defensible analytical data is supplied by subcontracted and government labs that support the various DOE and DOE Contractor Bioassay programs. Data quality objectives, which are project specific, will be addressed in site specific contracts via Contracts, Task Orders, laboratory Delivery Orders, etc. written against the Basic Order Agreement (BOA).**

## GENERAL BIOASSAY SERVICES

### INTRODUCTION

This Attachment to the Basic Ordering Agreement provides the technical requirements, analytical chemistry services, quality control requirements, and reporting requirements to provide radiochemical analysis of human urine, fecal, nasal swab, and tissue samples in support of DOE Contract Bioassay programs. The work will require various phases of radiochemical analysis including sample receipt; logging; tracking; sample preparation, separation, counting, results calculations; propagation of uncertainties, calculation of minimum detectable activity(MDA) and decision level(Lc); reporting and archiving of data; and quality control/quality assurance.

The Laboratory shall employ safe handling procedures, obtain the required licensing for analysis of Bioassay samples and for handling waste generated, utilize generally accepted good laboratory practices in the performance of contract requirements as specified in Attachment 1, “*Laboratory Analytical Services*” of the Basic Ordering Agreement. The Laboratory shall follow the quality assurance/quality control (QA/QC) program specified herein and as required in Attachment C, “*QA Program*” of the Basic Ordering Agreement.

The specifications in the Bioassay Requirements shall supersede any specifications found in the Basic Ordering Agreement in the case of conflicting statements.

# SUMMARY OF REQUIREMENTS FOR BIOASSAY

## 1. GENERAL REQUIREMENTS AND INFORMATION

This Attachment includes six sections that delineate the requirements for General Bioassay Support Services. The first section provides an overview of the general requirements for the bioassay task. The second section contains quality requirements specific to the Bioassay analytical work. The third section contains the specific analytical requirements for various methods related to instrumentation used for analysis and additional method specific QA/QC requirements. The fourth section is comprised of references and acceptable formulations for synthetic urine and synthetic fecal samples that may be used for matrix quality control samples. The fifth section defines the required data packages for Bioassay. The sixth section is an addendum to Attachment K of the Basic Ordering Agreement which consists of the bioassay analyte list with required detection limits and associated line item codes. When a term is used in the text without definition, the glossary (BOA Attachment G) meaning shall be applicable.

- 1.1. Privacy Act: The Information submitted to the Laboratory and the data produced by the Laboratory under this SOW are covered under the privacy act of 1974 and shall be protected from unauthorized access in accordance with applicable Federal law, 10 CFR 1008 Privacy Act and (Public Law 93-579)
- 1.2. Sample Characteristics
  - 1.2.1. The chemical form of the radionuclide(s) in the samples may be a metabolized product of normal biological processes, complexed as a result of combination with a chelating agent, or as a high fired oxide. The Laboratory shall use methods of sample preparation that assure dissolution of the entire sample, destruction of possible interferences for subsequent chemical separation, and that results, where necessary, in a sample where both tracer and analyte will undergo similar reactions during subsequent processing.
  - 1.2.2. Samples of urine may include either (1) the total simulated or real amount of urine excreted in 24 hours, or (2) a single or "spot" excretion. The samples are collected in plastic jars with plastic lids. The lids of all jars used should have a custody seal. If a sample jar is missing the custody seal or the custody seal is broken, the Laboratory shall record the deviation and contact the Site for further instructions and report the problem and its resolution in the data package Narrative.
  - 1.2.3. Samples of feces consist of a single excretion. The samples are collected in a plastic container with a plastic lid. The samples will be frozen prior to shipment and should have two (2) custody seals. If the custody seals are missing or broken, the Laboratory shall record the deviation and contact the Site for further instructions and report the problem and its resolution in the data package Narrative.
  - 1.2.4. Nasal/mouth swab samples consist of mucous from the mouth and nasal passages collected on cotton tipped swabs. The swabs will be packaged in new glass liquid scintillation vials that will be labeled with tape on the cap or enclosed within a plastic bag with the labeling on the bag. None of the glass surface of the vial should have any marking on it. The vials should be placed in a plastic bag and the custody seals applied to the plastic bag.

- 1.2.5. Tissue samples are collected on a gauze pad and sealed in a plastic bag. The gauze may also contain some blood. The entire sample, including the gauze pad shall be analyzed.
- 1.2.6. Samples submitted for Priority(21 day TAT), Rush(14 day TAT), or Rapid(7 day TAT) analysis should be assumed to involve quantities of radioactive material that may pose problems for cross-contamination. These samples shall not be processed with routine (no priority –30 day TAT) samples. Segregated laboratory areas are required.
- 1.2.7. Routine samples will constitute the bulk of the total analyses. Priority, Rush, and Rapid Analysis samples may be submitted periodically and are usually associated with a potential intake or other special occurrences.

***IT IS ESSENTIAL THAT ALL PRIORITY, RUSH AND RAPID SAMPLES SHALL BE ANALYZED AND REPORTED WITHIN THE SPECIFIED TURNAROUND TIMES. IMMEDIATE NOTIFICATION TO THE SITE IS REQUIRED WHENEVER FAILURE TO MEET TURN AROUND TIMES IS ANTICIPATED.***

- 1.2.8. At no time shall the Laboratory process different matrices or different priorities in the same batch.
- 1.3. **Work Performed Outside Scope of SOW:** Any additional analysis costs not addressed by the Basic Ordering Agreement including this Attachment shall be agreed upon in writing by the Site prior to the performance of the analysis or such costs will become the responsibility of the Laboratory.
- 1.4. **Radioactive Materials License:** The Laboratory shall meet the requirements contained in the Basic Ordering Agreement which include Attachment 1, Section 2.2.3.
- 1.5. **Analysis Capabilities:** The Laboratory shall maintain capability of performing analyses to the specified Required Detection Limit (RDL) as listed in the Bioassay Analyte Tables of this SOW.
- 1.6. **Subdivision of Priority, Rush, and Rapid Analysis Samples:** The Laboratory shall aliquot samples for priority, rush, and rapid determinations to ensure enough sample remains for a second analysis if required.
- 1.7. **Off-Normal Business Hours:** The Laboratory shall provide a point of contact available outside of normal business hours to implement emergency requests.
- 1.8. **Holiday List:** A list of subcontractor's recognized holidays shall be submitted at subcontract award and at any time the holiday listing changes.
- 1.9. **Notification Requirements:** The Laboratory shall meet the requirements of this Basic Ordering Agreement for sample protection, sample integrity, and late deliverables which include Attachment 1, Sections 3.1.2. and 3.1.3. In addition, the following notifications shall be made:
  - 1.9.1. *Results  $\geq 5 \times$  RDL:* The Laboratory shall provide immediate verbal and e-mail notifications to the Site no later than the close of the next business day for any sample results that exceed 5 times the RDLs specified in Attachment K. Written confirmation shall be provided to the Site no later than close of the next business day.
  - 1.9.2. *Nasal Smear Results Greater than Decision Level:* The laboratory shall provide immediate verbal and e-mail notifications to the Site no later than the close of the current business day for any nasal smear results which are greater than the decision level calculated for that sample. The Site will issue a current directive for a call list that will include three contacts and specific directions regarding failed contacts.



- 1.9.3. *Custody Seals*: The Laboratory shall provide immediate verbal and e-mail notification to the Site no later than close of the next business day following receipt of any samples received without custody seals or if the seals are broken.
- 1.9.4. *Weekly Status Reports*: The Laboratory shall provide weekly status reports to the Site with the following information:
- The number and types of samples received
  - The number and types of samples lost, missing, or destroyed
  - The number and types of analyses in progress
  - The number of recounts in progress
  - The number and types of analyses performed with results
  - Details of delinquent samples which have been in the laboratory for greater than the specified turn around time (TAT) without valid results and the expected delivery date of the data package.
  - Sample numbers for all samples on the status report.
- 1.9.5. *Blind Blanks*. The Site reserves the right to evaluate blind blanks shipped with samples when the reported results are positive and request written notification of the laboratory's investigation of the problem and corrective actions.
- 1.9.6. *Priority, Rush or Rapid Missed Turn a Round Time*. The laboratory shall provide written notification to the Site immediately when it is anticipated that turn around times for these samples will not be met.
- 1.10. **Recount Analysis**: The Laboratory shall perform recount analysis upon request by the site. A recount analysis shall require two counts except in the case where the first recount is above the decision level ( $L_c$ ) where only one count is necessary. The recount deliverable is a full sample data package as described in the Bioassay Data Package Components section of this attachment. All associated QC samples shall be recounted with the recount analysis request and be included with the sample data package.

## **2. FACILITY, INSTRUMENTATION AND KEY POSITION REQUIREMENTS**

- 2.1. **Facility**: The Laboratory facility shall meet all requirements of base analytical methods and the Laboratory Health and Safety Program specified in this Basic Ordering Agreement which include *Attachment 1, Section 2.2.8 and Attachment C, Section 6*.
- 2.1.1. The Laboratory shall maintain adequate facilities exclusively for low-level bioassay analyses. The Laboratory shall ensure that facilities and instruments are constructed, maintained and operated to emphasize radiological control to preclude the possibility of cross-contamination from radiological or environmental sources.
- 2.2. **Instrumentation**: The Laboratory shall have sufficient analytical equipment and capability to meet all terms and conditions of this Basic Ordering Agreement, including all equipment requirements specified in base methods used to perform the analyses.
- 2.2.1. At a minimum, the Laboratory shall have operational instrumentation necessary for the proposed work at the time of the on-site evaluation. This instrumentation shall be committed for the full duration of the contract and capable of producing data as described herein.
- 2.3. **Materials**: Materials shall be of the quality and capability necessary to meet the requirements of this Basic Ordering Agreement.

- 2.4. **Key Position Requirements:** The Laboratory shall assign individuals the responsibilities for the Bioassay technical key positions listed below in addition to the key positions listed in the BOA.. Personnel Training and Qualification Requirements of the BOA Attachment B shall apply with the following minimum academic training and experience qualifications for each of the Bioassay technical key positions. A qualifying individual may fill more than one position.

2.4.1. *Radiological Counting Room Specialist*

Responsibility: Responsible for the proper operation, maintenance, calibration and data processing for all necessary instrumentation.

Academic Training: A minimum of a bachelor's degree in chemistry or physics.

Experience: A minimum of two years of experience in the operation, maintenance, and data processing for counting room instrumentation. This will include formal vendor supported or accredited training or like experience.

2.4.2. *Technical Lead/Supervisor*

Responsibility: Responsible for radiochemistry methodology as related to the scope of work outlined in this SOW. Responsibilities also include technical review for acceptability of all data produced for this SOW.

Academic Training: A bachelor's degree in chemistry with emphasis in analytical chemistry.

Experience: A minimum of five years of work experience in analytical radiochemistry to include radiochemical separation methodology, techniques, and technical supervisory responsibility. The responsible supervisor shall also have a working knowledge of counting instrumentation, the data produced, and all pertinent radiochemical calculations

## QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

This Attachment requires a variety of activities that represent the minimum quality assurance/quality control (QA/QC) operations necessary to satisfy analytical quality requirements. These operations and those in the Basic Ordering Agreement, Attachment C are designed to ensure that data produced meets defined minimum data quality objectives. These requirements do not release the Laboratory from maintaining its own QC checks on method and instrument performance.

### 1. QUALITY ASSURANCE PLAN

Requirements for the quality assurance plan are specified in the Attachment C, QA Program of the Basic Ordering Agreement.

### 2. ANALYTICAL BATCH QUALITY CONTROL REQUIREMENTS

A batch of samples is 10 samples or less plus the minimum QC samples consisting of a batch blank and a laboratory control sample. As described in Sections 5.3 and 5.4 below, the batch may also consist of a duplicate and/or a matrix spike. A batch of samples is processed throughout the entire analytical process together. If equipment restrictions limit the number of samples in any particular step, the samples in the batch shall be processed continuously and consecutively until the entire batch is completed.

Each ten Site samples shall have a matrix blank and LCS uniquely associated with them that shall pass data quality objectives for those ten sample results to be valid.

*[COMMENT: We do know the standard batch size for environmental is 20 samples. Because of all of the possible QC failures, at RFETS we have limited the batch size to 10 samples so that, for example, if the batch LCS fails (relative bias, etc.), only 10 and not 20 samples will fail] . This can be a Site specific requirement if we cannot reach agreement. Also environmental batches of 20 samples require not only a batch blank and LCS but also a duplicate sample and a matrix spike – so either way you have 2 QC for 10 samples or 4 QC for 20 samples.]*

Duplicates and Matrix Spikes are not common in Bioassay analyses because of RDL requirements. It is possible that the entire sample must be used in one analysis in order to meet the RDL or to meet as low an RDL as possible – thus it is often the case that there is not sufficient sample for a sample duplicate or a matrix spike. Analysis of duplicate samples and/or matrix spikes is a Site specific requirement.

*[COMMENT: Site Specific requirements will have to address the use of duplicates and matrix spikes. If they are not a part of the routine required QC, they will most likely have to be billed as batch samples.]*

- 2.1. **Sample Control:** All laboratory identifications(ID) used for the prepared samples through the entire analysis (e.g. identifications of beakers, planchets, filter papers, vials, or their holders, etc.) shall be documented and traceable to the Site sample identifications and the respective preparation Analytical Batch Identification.

- 2.1.1. The Analytical Batch ID for customer requested reanalysis/recount shall be different from that of the original analysis. The exact form of the identification may be defined by

the laboratory as to whether the ID shall be completely different or may have the original ID with an extension. All reanalysis shall be traceable to the reanalysis Analytical Batch Identification and to the reanalysis Analytical Batch QC samples.

- 2.2. **QC Sample Identification:** All QC samples in the Analytical Batch shall be identifiable as to QC type (e.g., Batch Blank, Laboratory Control Sample, Duplicate, and Matrix Spike) and shall be traceable to the preparation Analytical Batch identification.
- 2.3. **QC Sample Preparation:** All samples and QC samples in each Analytical Batch shall be prepared concurrently and in the same manner.
- 2.4. **QC Sample Counting:** All QC samples shall be counted and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame and using the same instrument calibration parameters, instrument analysis algorithms, etc.
  - The same time frame implies that where multiple detectors are used and are sufficient to count the entire batch at the same time, the entire batch may be counted at the same time. If the number of detectors is not sufficient to count the entire batch at the same time, then samples shall be counted consecutively on the available detector(s).
  - The same instrument calibration parameters, instrument analysis algorithms, etc. implies that these parameters for a given instrument shall not be changed for the samples in that batch. It is understood that for multiple detectors, the parameters may not be identical.

### 3. QC MATERIAL TRACEABILITY

- 3.1. All tracer, carrier, matrix spike, and LCS aliquots shall be traceable to their respective primary standard reference material (SRM) certificate (if applicable, minimally to the reagent lot number and supplier, the standard log, and the respective preparation Analytical Batch ID).
- 3.2. All QC materials shall have identified expiration dates (lab assigned for all preparations and any stock materials with no expiration dates provided by the supplier). No QC materials shall be used beyond their expiration dates. Results associated with expired quality control materials are not valid.
- 3.3. Tracer, LCS, and matrix spike materials shall be prepared from the National Institute of Standards and Technology (NIST) traceable standards. If a NIST traceable standard reference material cannot be procured, then the standard shall meet the requirements for a “working reference material” as described in STD.ASTM C1128.

### 4. TRACEABILITY OF MEASURING AND TESTING EQUIPMENT (M&TE):

All pipet and balance identifications specified on all Analytical Batch preparation benchsheets or logs shall be traceable to the respective calibration log.

### 5. BATCH QUALITY CONTROL SAMPLES

- 5.1. **Batch Blank:** The batch blank is a laboratory-generated sample prepared with absence of the analyte of interest. Batch blanks are batch quality indicators and are carried through the entire sample analysis procedure with the samples in the batch.
  - 5.1.1. **Matrix:** Batch blanks shall have a matrix and sample size similar to the actual samples being analyzed. Acceptable synthetic matrices for urine and fecal samples are given in Section 4 “References” of this Attachment. The minimum acceptable volume for a batch blank for routine urine analysis(24 hour void) is 1200 mls. The minimum

acceptable weight for a batch blank for fecal samples from the synthetic formulation given in Reference Section is 65 grams.

5.1.2. **Frequency:** One batch blank shall be prepared and analyzed with every Analytical Batch of samples.

5.1.3. **Counting:** Batch blanks shall be counted for a sufficient time to meet the required detection limit except in the case where the achieved MDA and Lc are calculated from the standard deviation of a blank population. In this case the batch blanks shall be counted for the same count time as the samples.

5.1.4. **Acceptance Criteria:**

- The MDA of the batch blank shall be less than the RDL unless all samples in batch are positive, as defined by the Site.
- If all sample results in the batch are greater than the RDL, then the Batch Blank MDA shall be less than the activity of the least active sample in the batch of that sample.
- If all of the samples in the batch are less than the RDL, the activity of the blank shall be less than the MDA.
- The laboratory may wish to further define limits on the activity of batch blanks such that MDAs are achievable when blank populations are used to calculate MDA.
- Refer to Section 9 for reanalysis requirements.

5.2. **Laboratory Control Sample (LCS)** The laboratory control sample is a quality indicator and provides information about the relative bias of the analysis. It is used to assess the overall process for any inherent biases or trends. The LCS contains known quantities of analyte and is carried through the entire analysis procedure with the samples.

5.2.1. **Frequency:** At least one LCS shall be prepared and analyzed with every Analytical Batch of samples.

5.2.2. **Selection and Level:** The LCS shall be of the same analyte(s) as the sample analyte(s) and shall be at least 5 times but not greater than 20 times the RDL with the following exceptions:

- For RDLs of low activity the analyte shall be at a level where the random counting error does not exceed 10% in the counting time required to attain the RDL.
- Analytes for gamma spectroscopy need not be the same as the sample analyte(s) but should fall in the approximate energy region of the spectrum (low, mid-range, or high energy) as the analyte(s).
- For gross alpha, gross beta analysis, Site specific guidance will be provided for selection of the analyte and acceptable relative bias when the analyte differs from that used for the calibration curve.

5.2.3. **Counting:** The LCS shall be counted for a sufficient time to meet the required detection limit.

5.2.4. **Matrix:** All batch LCS shall be matrix LCS having a matrix, sample size and other relevant characteristics similar to the actual samples in the batch. Acceptable synthetic matrices for urine and fecal samples are given in the "References" section of this Attachment. The minimum acceptable volume for a batch LCS for routine urine analysis(24 hour void) is 1200 mls. The minimum acceptable weight for a batch blank

for fecal samples from the synthetic formulation given in the References section of this Attachment is 65 grams.

5.2.5. Acceptance Criteria:

The relative bias as calculated from the formula:

$$\text{Relative bias} = \frac{\text{observed} - \text{known}}{\text{known}}$$

The relative bias shall be in the range -.25 to +.25. (Reference: ANSI N13.30, Appendix B)

*COMMENT: We can make the upper limit +50% to correspond exactly with ANSI if it is wanted by a majority.*

- For gross alpha, gross beta analysis, the acceptance criteria are applicable when the analyte in the LCS is the same analyte used for the calibration curve. Site specific instructions will be provided when the LCS analyte is different from that used for the calibration curve.
- Site/Project specific requirements may be provided.
- Refer to Section 9 for reanalysis requirements.

5.3. **Duplicates:** The purpose of the Duplicate sample analysis is to assess the Laboratory precision by providing information on the Laboratory's reproducibility of results, and the homogeneity of the sample. The Duplicate activity shall not be averaged with the corresponding sample activity when reporting results.. The Site may require that a specific sample be used for Duplicate sample analysis. Duplicates are not common in Bioassay analyses because RDL requirements may preclude the use of aliquots. Laboratory precision may be assessed by calculating the precision of batch matrix laboratory control samples(LCS).

*COMMENT: EXAMPLE from RFETS SOW: The precision shall be determined initially for at least 5 LCS samples and updated thereafter using up to the last fifteen(as many as are available up to 15 with analysis dates not more than 6 months old). We can include some such requirement if a consensus for it can be reached. Please include in your comments what your preference is.*

**Frequency:** The frequency of duplicates will be determined by Site specific requirements.

5.3.1. **Counting:** The duplicate shall have the same count time as the original sample. If LCSs are used to assess precision rather than duplicate sample analysis, all LCs in a given precision calculation shall have the same count time.

5.3.2. **Precision Assessment:** The relative precision shall be calculated as described in ANSI N 13.30, Section 4.3.3. The calculated precision shall be less than or equal to 40%.

5.4. **Matrix Spikes:** Matrix spikes consist of analysis of a replicate of an actual sample to which a known quantity of the analyte has been added. Recovery (determined as the percentage of "found" analyte relative to the known amount introduced) provides information on sample specific matrix effects that result in an analytical bias for a given analysis batch. (e.g. H-3, C-14, etc.) Matrix Spikes shall be added as early in the sample preparation steps as practicable. Matrix spikes are not common in Bioassay analyses because of RDL requirements. It is possible that the

entire sample must be used in one analysis in order to meet the RDL or to meet as low an RDL as possible – thus it is often the case that there is not sufficient sample for a matrix spike.

- Matrix spikes are not required for radiochemical analyses if an isotopic tracer or chemical carrier is used in the analysis to determine chemical recovery (yield) for the chemical separation and sample mounting procedures. Matrix spikes are not required for Gross Alpha, Gross Beta, or Gamma Analysis.
- Matrix spikes shall be run on a separate sample aliquot using the same analyte as that being analyzed whenever possible.

5.4.1. **Frequency:** The frequency of matrix spikes will be determined by Site specific requirements.

5.4.2. **Selection and Level:** The matrix spike shall be added at a concentration of at least 5 but not greater than 20 times the RDL. In samples having known significant activity of the radionuclides to be analyzed, more than 20 times the RDL may be added to minimize the effect of the sample activity on determination of spike recoveries.

5.4.3. **Counting:** The matrix spike shall be counted for a sufficient time to meet the required detection limit.

5.4.4. **Acceptance Criteria: Matrix** spike recoveries shall be within the control limits of 60 – 140%. Matrix spike samples for which the sample activity is greater than five times the spiking level are not required to meet this criteria.

5.4.5. Refer to section 9 for reanalysis requirements.

## 6. RECOVERY OF TRACERS AND STABLE CARRIERS

Isotopic tracers are typically radioactive materials (e.g., Pu-242, Sr-85) while carriers are typically nonradioactive (e.g., natural strontium). They are added to samples to determine the overall chemical yield for the analytical preparation steps. When tracers or carriers are used, each sample (including any batch associated QC samples) shall be “spiked” separately with the same materials and individual sample yields will be determined. The tracer/carrier shall be added to the sample at the very beginning of the sample preparation procedure.

6.1. **Isotopic Tracers:** The recovery of isotopic tracers shall be in the range 30% - 110%.

6.2. **Stable Carriers:** The recovery of stable carriers shall be in the range 40% - 110%.

6.3. **Reanalysis Requirements:** Refer to Section 9 for reanalysis requirements.

## 7. RESULTS REPORTING REQUIREMENTS

7.1. **Reporting Figures:** All reported quantities including but not limited to result, total propagated uncertainty, and minimum detectable activity shall be reported to three digits in scientific notation.

7.2. **Negative Numbers:** All negative activities shall be reported as such. If the sum of the activity and the measurement uncertainty at 3-sigma is a negative number, the sample shall be re-counted before it is reported and the problem documented in the Case Narrative. Recurrent problems with significant negative results suggest that the background subtraction and/or blank subtraction may be in error or that the estimate of error is low.

7.3. **Total Propagated Uncertainty:** All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the total propagated uncertainty of a result shall be

documented in an appropriate laboratory SOP. The total propagated uncertainty shall include both systematic and random error.

7.3.1. **Systematic Error** shall include but is not necessarily limited to:

- the errors from all measurement devices such as but not limited to pipets and balances.
- the uncertainty of known values of tracer solutions, calibration uncertainties, uncertainty about the mean blank value when blank subtraction is done, etc.

7.3.2. **Random Error** shall include but is not necessarily limited to the total random counting error associated with each sample and appropriately propagated when more than one variable is used to determine the result.

## 8. MINIMUM DETECTABLE ACTIVITY (MDA) and DECISION LEVEL(Lc)

8.1. **The MDA (minimum detectable amount)** is the smallest amount (activity or mass) of an analyte in a sample that will be detected with a probability  $\beta$  of non-detection (Type II error) while accepting a probability  $\alpha$  of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). For the purposes of this SOW, the  $\alpha$  and  $\beta$  probabilities are both set at 0.05 unless otherwise specified. (Reference: *ANSI N13.30 and ANSI N42.23*)

8.2. **The Lc (decision level or critical level)** is the quantity of analyte at or above which a decision is made that the analyte is definitely present. The decision level shall be calculated as the *a posteriori* measurement level at which activity is significantly above background (with 95 percent confidence except for gross alpha in nasal swabs where the confidence level is 99%) using the method defined in ANSI N13.30.

8.3. **MDA and Lc Factors and Conditions:** MDAs and Lcs are determined based on the factors and conditions, which influence the measurement. Sample size, count duration, tracer recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the RDLs. If RDLs are not achieved, then the cause shall be addressed comprehensively in the Case Narrative.

### 8.4. MDA and Lc Calculation

8.4.1. Site specific requirements will be provided for implementation of MDA and Lc calculations.

8.4.2. Use of blank populations for calculation of MDAs and decision levels(Lc) shall be defined in a laboratory SOP which at a minimum addresses:

- The actions required to administratively control the assignment of blanks to the blank population.
- The number of blanks to use in the population..
- How the blank population changes.
- The actions required to administratively control the removal of a blank from the blank population.
- How matrix batch blanks will be used when only an aliquot of the blank is used for analysis, i.e. how will the mean blank value for blank subtraction be determined and what will be used in the blank population to determine the standard deviation. In addition, the case of the entire blank being analyzed but only aliquots of samples are analyzed shall be similarly addressed.



- When blank populations are used, the appropriate blank subtraction shall be the mean blank value of the blank population.
  - The procedure for blank populations shall address blank subtraction when the mean blank value is negative.
  - The method of implementation shall not introduce any statistical bias
- 8.4.3. The implementation of blank populations for calculation of MDAs and Lcs shall be described in detail in an SOP, which is accepted by the Site prior to use.(Reference: *ANSI N13.30*, Appendix A.5.13 for examples of MDA calculations)
- 8.5. **MDA for Results Significantly Greater than RDL:** The laboratory shall optimize analysis parameters in order to achieve analyte MDAs less than or equal to the RDLs, except when sample activities are significantly greater than the RDL. Samples with elevated activities shall be handled according to the following requirements:
- 8.5.1. The appropriate aliquot size shall be determined based on the activity level in the sample. The aliquot shall be large enough to generate data, which meet the following criteria:
- The measurement uncertainty shall not be greater than 10% of the sample activity.
  - The MDA for the analysis shall be a maximum of 10% of the sample activity.

## 9. CONDITIONS REQUIRING REANALYSIS

If reanalysis is not possible, the Site shall be contacted for specific guidance/requirements.

### 9.1. General Conditions

9.1.1. If the RDLs could not be achieved because of laboratory errors or oversights such as inadequate count times, inadequate aliquot size, inappropriate dilution, low detector efficiencies, high detector backgrounds, etc., then the sample shall be reanalyzed/recounted under more optimal conditions.

### 9.2. Sample and Analyte Specific Conditions: Any one of the following are additional conditions that require reanalysis for a particular sample and analyte when sufficient sample is available:

9.2.1. If for any reason sample or batch QC integrity becomes suspect (e.g., spillage, mis-identification, cross-contamination), all potentially affected samples shall be reanalyzed from a point before that which the integrity came into question. If new batch QC must be prepared for reanalysis, samples for reanalysis shall be restarted at the normal point of initiation for the batch QC.

9.2.2. All samples failing the criteria for tracers or carriers as defined in Section 6.

9.2.3. All samples associated with expired standards.

9.2.4. If there is not sufficient sample for re-analysis, the sample data described above is failed data. The reasons for failure shall be documented in the Case Narrative.

### 9.3. Analytical Batch Conditions: Except where noted otherwise, any one of the following conditions requires reanalysis of the entire Analytical Batch, beginning with the preparation when adequate sample is available:

9.3.1. Batches, which failed the Batch Blank criteria as defined in Section 5.1.4.

9.3.2. Batches, which failed the LCS criteria as, defined in Section 5.2.5.

9.3.3. Batches, which failed the Matrix Spike criteria as, defined in Section 5.4.4.

9.3.4. If there is not sufficient sample for re-analysis, the sample data in batches as described above is failed data. The reasons for failure shall be documented in the Case Narrative.

**9.4. CONDITIONS REQUIRING A RE-COUNT**

9.4.1. If the RDL was not achieved due to inadequate count duration, low detector efficiencies, or high detector backgrounds, the sample shall be re-counted under more optimal conditions, and the reasons for the re-count shall be documented in the Case Narrative.

9.4.2. Any re-counts of either the blank or the LCS require a re-count of the entire associated batch. Any re-counts of samples necessitate a re-count of the sample placed in a batch with the associated batch blank and LCS.

9.4.3. Additional Site specific requirements may be provided for re-counts.

**10. VERIFICATION OF STANDARDS PREPARATIONS:**

In addition to the general requirements of this Basic Ordering Agreement pertaining to all standards which include Attachment C, Criterion 8 the following requirements for verification of prepared standards shall be observed.

10.1. Standards shall be verified prior to initial use.

10.2. Preparations of standards solutions used for a period of time exceeding one year shall be verified annually, at a minimum, and documented in a logbook.

10.3. At least three verification measurements of a standard shall be used to determine the mean value and standard deviation of the verification results.

10.4. The certificate value (NOT including any uncertainty) shall lie within the 95% confidence interval determined from the mean and two sigma standard deviation of the three measurements.

10.5. The two sigma value used for the 95% confidence interval shall not exceed 10% of the mean value of the three verification measurements.

**11. MEASURING AND TESTING EQUIPMENT REQUIREMENTS**

Requirements for measuring and testing equipment are specified in Attachment C of this Basic Ordering Agreement.

**12. DATA MANAGEMENT**

Requirements for Site data management are specified in Attachment 1, Section 3.1.6 and Attachment C of this Basic Ordering Agreement.

**13. PERFORMANCE EVALUATION (PE) SAMPLES**

13.1. Performance Evaluation Program Participation

13.1.1. **Required Analytes:** The Laboratory shall participate in an interlaboratory comparison study that includes each analyte, matrix and method used to report Site samples when such interlaboratory comparison samples are available. Participation in interlaboratory comparison studies for methods used for sample screening is not required.

13.1.2. **Frequency:** The minimum required frequency of participation in inter-laboratory comparison studies is semi-annually.

13.1.3. **Acceptable PE Programs:** Participation in one or more of the following evaluation programs are acceptable under this statement of work.

- Bioassay Intercomparison Study, Oak Ridge National Laboratory
- DOELAP Radiobioassay In-Vitro Pilot Study
- Procorad – this is an international program with headquarters in France

13.1.4. **Unacceptable Performance:** Reporting an unacceptable value falling outside the warning limits, as calculated by the program, will result in a probationary period until the next reporting period for that analyte. If the Laboratory fails two consecutive evaluations, the Laboratory will not receive samples for analysis by the failed method until an acceptable PE score has been achieved or other verification of corrective action. Root cause and corrective action reports for PE samples outside of acceptable limits are to be submitted to the potentially affected Site(s) within 21 days from receipt of the scores.

13.1.5. **PE Score Disclosure:** PE scores and names of laboratories under this SOW will be available to the DOE and any other appropriate organization and/or individual procuring analytical services for DOE.

## **14. LABORATORY EVALUATION SAMPLES**

Prior to award of contract, laboratories shall be tested with a minimum of seven test samples consisting of at least two blanks and at least five control samples. Any change in scope of the contract or when a laboratory is inoperable for a given analysis for more than 30 consecutive calendar days will require testing with evaluation samples.

14.1.1. These samples shall be evaluated by the Site using the requirements of this SOW for data and data packages. The major criteria for evaluation shall be MDA, relative bias, average relative bias and relative precision as specified in this SOW.

## **15. INSTRUMENT MAINTENANCE/REPAIR DOCUMENTATION**

- 15.1. The laboratory shall identify and document the instrument manufacturer, model number, configuration, settings, detector identifications, and any modifications in the instrument maintenance log.
- 15.2. All repairs and modifications shall be documented.
- 15.3. Following all repairs and modifications, verification of calibration and background determination and/or calibration with background determination shall be performed and documented.

## **16. ON-SITE LABORATORY EVALUATIONS**

Requirements for on-Site laboratory evaluations are specified in Attachment B, Initial/Continuing Laboratory Assessment of this Basic Ordering Agreement.

# RADIOANALYTICAL METHODS

## 1. METHOD SELECTION AND APPLICATION

This section contains general method requirements for analytical radiochemistry that are applicable to all radiochemical analysis. The requirements for specific analyses shall supersede any general requirements in the case of conflicting statements. General requirements applicable to all samples and not just to radiological samples are given in Attachment 1 of this Basic Ordering Agreement.

### 1.1. **Selection of Method:** Methods used for analysis shall meet the following requirements:

- 1.1.1. Methods shall meet the required detection limits for bioassay samples as given in the Bioassay Analyte Tables of Attachment K.
- 1.1.2. Analytical methods selected to produce data to meet the requirements of this SOW shall not have conditions and limitations that can preclude the possibility of meeting the data requirements. This condition applies to sample preparation, separation, preparation for counting, and actual counting or measurement of the sample.
  - The analytical method selected shall be capable of producing data that meets the minimum method QA/QC requirements. All exceptions shall be accepted by the Site prior to use.
  - Methods selected and subsequent SOPs shall be tested and validated with matrix control samples and matrix blanks prior to analysis of Site samples.

## 2. SAMPLE HOLDING TIMES AND PRESERVATION REQUIREMENTS

- 2.1. **Holding Time:** The maximum sample holding time allowable under this contract is 180 days. In addition the maximum sample holding time shall not exceed five half-lives of an unsupported nuclide (see Glossary definition, Attachment G) of interest when five half-lives is shorter than 180 days. Sample specific guidance/requirements may be provided by the Site for specific isotopes or for very short-lived isotopes.
- 2.2. **Preservation Requirements:** General Bioassay Samples are not preserved before shipment to the laboratory. Laboratory sample preparation methods shall address any problems that could occur because of lack of preservation. If samples are held before any preparation is done such that the turn around times cannot be met, the Site shall be notified prior to sample pour-up. It is the prerogative of the Site at such time to provide specific guidance/requirements regarding sample pour-up and rinsing of the sample containers. All such issues shall be reported in the Case Narrative.

## 3. SAMPLE ALIQUOTS AND UNUSED SAMPLE PORTIONS

- 3.1. **Aliquots:** The Laboratory shall aliquot priority, rush, and rapid samples to insure that enough sample remains to provide a second analysis if required. Urine samples for priority, rush, and rapid analysis shall be subdivided after acid is added and samples have been removed from the original container. Fecal samples shall be subdivided after total dissolution of the samples. The laboratory shall assure that all aliquots taken for analysis are representative of the entire sample. The laboratory shall assure that any sample-to-sample contamination during preparation, separation, and counting is eliminated. The laboratory shall store and preserve the integrity of the unused sample portions or preparations during all phases of processing.

- 3.2. **Unused Sample Portions:** Unless otherwise specified by the Site, unused sample portions, dissolved samples, and counting preparations shall be maintained for a minimum period of 90 calendar days after receipt of the analytical results by the Site. This unused sample portion allows for a possible sample rerun/recount if needed.

#### **4. RADIOLOGICAL CONTROL**

The laboratory shall maintain a radiological control program that rigorously addresses analytical radiological control. The program shall address the procedures for segregating samples with potentially widely varying levels of radioactivity. The radiological control program shall explicitly define how low level and high level samples will be identified, segregated and processed in order to prevent sample cross-contamination.

#### **5. WATER PURITY**

Water purity shall be at least distilled or deionized water, or as specified on a case-by-case basis by the Site. Further water purity guidance/requirements are found in the analysis specific requirements of this SOW.

#### **6. ISOTOPIC DETERMINATIONS BY ALPHA SPECTROMETRY**

##### **6.1. Internal Tracer Method**

- 6.1.1. The internal tracer method shall be used for isotope specific analysis by alpha spectrometry. All exceptions shall be Site accepted prior to use.
- 6.1.2. The tracer shall be added to the sample at the very beginning of the sample preparation procedure.
- 6.1.3. Initial sample preparation shall include treatment to ensure that tracer and analyte will undergo similar reactions during processing.
- 6.1.4. All tracers used for alpha spectrometry shall be tested by the lab for contribution in the ROIs of the analytes of interest. If a significant contribution is found, the method for correction shall be Site accepted prior to use.

##### **6.2. Background Correction**

- 6.2.1. The gross counts in each analyte and tracer region of interest (ROI) shall be corrected for the particular detector's background contribution in those same ROIs.
- 6.2.2. Site/Project specific instructions may be provided regarding the required background subtraction.

##### **6.3. Blank Correction**

- 6.3.1. Blank correction shall be routinely done for sample results. Batch blanks shall not be blank subtracted. Site specific instructions will be provided for the implementation of blank correction. All blank correction requirements will be communicated to the laboratory prior to award of contract or contract modification.

##### **6.4. Conditions Requiring Reanalysis**

- 6.4.1. **Sample and Analyte Specific Conditions:** Any one of the following are additional conditions that require reanalysis for a particular sample and analyte.

- 6.4.1.1. If the tracer recovery for the sample does not fall within 30% - 110%, reanalysis is required, beginning with preparation.
- 6.4.1.2. If the full width half maximum (FWHM) for the tracer peak exceeds 100 keV and/or the peak energy does not fall within  $\pm 50$  keV of the known peak energy, reanalysis is required. If the problem is related to the detector, a re-count on another detector may be acceptable. If the problem is related to inadequate sample preparation, then reanalysis is required.
- 6.4.1.3. If the analyte and tracer peaks are not resolved because the analyte activity is significantly larger than the tracer activity, the sample shall be re-analyzed with a smaller aliquot such that resolution of tracer and analyte peaks is accomplished. Site specific guidance may be provided on a case by case basis for adequate resolution of tracer and analyte peaks.
- 6.4.1.4. If the sample analyte spectrum contains significant interferences with the analyte and/or tracer ROIs, reanalysis is required. Site specific guidance may be provided on a case by case basis for significant interference.
- 6.4.2. **Analytical Batch Conditions:** Except where noted otherwise, any one of the following additional conditions requires reanalysis of the entire Analytical Batch, beginning with the preparation:
- 6.4.2.1. If the tracer chemical recovery for the Batch Blank does not fall with 30% - 110%, reanalysis is required if sufficient sample is available.

## 6.5. Instrument Calibration

The purpose of the instrument calibration is to ensure that alpha spectrometry detectors used for sample analysis are capable of producing quality results according to the specifications given in this section, and that the calibration was maintained throughout the time period in which samples were analyzed.

- 6.5.1. **Calibration:** Calibration of each alpha spectrometry detector used to produce data for this SOW shall include:
- channel vs. energy calibration,
  - efficiency determination
  - background determination for each ROI.
- 6.5.2. **Frequency of Calibration**
- 6.5.2.1. Channel vs. energy calibration shall be done at least monthly.
- 6.5.2.2. Background determinations for each ROI shall be done at least monthly.
- 6.5.2.3. Actual efficiency determinations shall be performed when a new detector is put in service and/or when the check source count is outside of the acceptable limits of the control chart (Reference: *ANSI N42.23*, Annex A5). Check source counts shall be done at least monthly.
- 6.5.2.4. Calibration for energy and background determination and efficiency determination shall be performed when a new detector is put into service or if repair is performed on an existing detector.
- 6.5.2.5. Site specific requirements may be provided.

### 6.5.3. Calibration Standards

- 6.5.3.1. Efficiency determinations shall be performed with sources, which are themselves NIST traceable or with sources prepared from NIST traceable standards.
- 6.5.3.2. When sources used for determinations of efficiency are prepared from NIST traceable standards, they shall be “working reference materials” as defined in *STD.ASTM C1128*. A material balance check shall be done on each source which clearly demonstrates that greater than 99% of the standards used were carried on the source. The material balance check shall be done on the fraction remaining from either the neodymium fluoride precipitation or the electrodeposition plus all rinses from an adequate cleaning of any vessel used in the process. The estimated error in preparing the source shall be propagated into the error of the efficiency determination.
- 6.5.3.3. Check sources shall be used only to verify that efficiencies have not changed. They shall not be used to determine efficiencies.

### 6.5.4. Energy Calibration Requirements

- 6.5.4.1. A curve shall be fit for Energy (Y-axis) versus Channel (X-axis), and the equation with the slope and Y-intercept for the fit shall be documented.
- 6.5.4.2. The slope of the equation shall be <15 keV/channel.
- 6.5.4.3. The energy calibration shall be performed using at least three isotopes within the energy range of 3 to 6 MeV.
- 6.5.4.4. The final peak energy positions of all observed isotopes shall be within  $\pm 40$  keV of the expected peak energy.

### 6.5.5. Background Requirements

- 6.5.5.1. The Background total counts (or counts per unit time) for each target analyte and tracer isotope ROI shall be analyzed on each detector and documented.
- 6.5.5.2. The Background for each ROI shall be sufficiently low such that RDLs can be attained.
- 6.5.5.3. The limits of acceptability for each background ROI shall be documented. These shall be set such that RDLs can be obtained for backgrounds at the limit of acceptability.
- 6.5.5.4. Background count times shall be equal to or longer than sample count times.

### 6.5.6. Efficiency Determination Requirements

Detector efficiency is not used in the calculation of results when tracers are used in the analysis, but only used to calculate the estimated yield, which is used as a general method performance indicator and in some implementation of MDA and Lc calculations.

- 6.5.6.1. The Efficiency counts for the ROI shall be background corrected using the same ROI for the background unless the background is less than 0.5% of the total counts in the ROI.
- 6.5.6.2. The Efficiency shall be determined on at least 10,000 net counts in the ROI (after background correction).

6.5.6.3. Check source counts to verify efficiency shall be determined on at least 2,000 counts.

6.5.6.4. The Efficiency and Efficiency error shall be documented.

6.5.6.5. The efficiency check as determined by the check source count and its associated error and limits of acceptability for the check source result shall be documented.

## 6.6. **Spectrum Assessment**

6.6.1. ROIs shall be clearly indicated either graphically or in tabular form on alpha printouts.

6.6.2. The FWHM resolution for each sample and QC sample tracer peak shall be  $\leq 100$  keV.

6.6.3. The tracer peak energy for each sample and QC sample shall be within  $\pm 50$  keV of the expected.

6.6.4. Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptable spectral resolution, acceptable energy calibration and interferences with the analyte and tracer ROIs.

## 7. **LIQUID SCINTILLATION COUNTING**

### 7.1. **SAMPLE PREPARATION**

7.1.1. **Tritium in Urine:** Unless Site specific requirements are provided, urine samples for tritium analysis and all associated QC samples shall be distilled prior to analysis. The applicable preparation SOP shall specify the fraction to be collected. The same fraction shall be collected for samples and all associated QC samples.

7.1.2. **Other:** (e.g. C-14, Fe-55, Ni-63, Tc-99, I-129, I-131, Pm-147, Pb-210, Pu-241) Due to the variety of effective methods for the analysis of various matrices and analytes, specific sample preparation procedures shall be accepted by the Site prior to use.

7.1.3. **Gross Alpha for Nasal Swabs:** Sample preparation SOPs shall be Site accepted prior to use. Site specific requirements shall be provided.

### 7.2. **Counting Vial Preparation**

7.2.1. Samples shall be counted in vials equivalent to or superior to low potassium glass vials or high density polyethylene vials specifically manufactured for liquid scintillation counting. Samples in polyethylene vials shall be counted within a time period not to exceed the manufacturer's specification for the cocktail used in the analysis.

7.2.2. Vials shall be prepared according to manufacturer's specification for the cocktail. The vials shall be "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting. The prepared vials shall be inspected to verify that the sample loaded properly in the cocktail.

### 7.3. **Instrument Calibration**

7.3.1. **Calibration Procedures:** Calibration procedures for liquid scintillation counters shall incorporate and adhere to ANSI N42.15-1997, *American National Standard Check Sources for and Verification of Liquid Scintillation Systems*. References are for the customer determined current version. When references change, an implementation schedule will be determined.



- 7.3.2. **Instrument Set-Up:** The instrument shall be set up either by the manufacturer or according to the manufacturer's instructions. Any deviations shall be documented in the instrument maintenance log.
- 7.3.3. **Non-routine calibration frequency**
- 7.3.3.1. The instrument shall be calibrated whenever check source data from Instrument Performance Assessment indicates that the instrument performance may have changed.
- 7.3.3.2. The instrument shall be calibrated whenever instrument repair or maintenance that may have affected counting performance is done.
- 7.3.4. **Routine calibration frequency**
- 7.3.4.1. Instrument backgrounds shall be determined daily or with each batch of samples.
- 7.3.4.2. Quench curves for external standardization shall be generated at least yearly.
- 7.3.4.3. For constant quench calibration, the efficiency standards shall be counted weekly or with each counting batch.
- 7.3.5. **Instrument Background**
- 7.3.5.1. The instrument background vial shall be prepared with low-tritium or "dead" water. The instrument background vial shall be prepared with the same water to cocktail ratio as the samples are prepared.
- 7.3.5.2. The type of water used to prepare the instrument background vial shall be explicitly noted on the preparation and counting documentation.
- 7.3.5.3. The instrument background shall be determined with each sample batch. The most recent background count shall be used to calculate sample activities and MDAs.
- 7.3.6. **Efficiency**
- 7.3.6.1. For analysis methods using quench curves to determine individual sample counting efficiency, the quench curves shall be generated at least yearly and verified after any instrument maintenance.
- 7.3.6.2. If the calibration method is constant quench, the efficiency standards shall be counted weekly or with each counting batch.
- 7.3.7. **Instrument Performance Assessment(IPA):** This check shall be done daily on days when samples are counted.
- 7.3.7.1. It shall include at a minimum a background count and for use of the beta region of interest, two check source counts using H-3 and C-14 . For use of the alpha region of interest only the check source count shall be done using an alpha emitter.
- 7.3.7.2. All daily IPA measurements shall be control charted with the daily values falling within 3s of the mean of at least the last ten measurements.
- 7.4. **Additional Data Assessment**
- 7.4.1. **Sample Specific Conditions:** In addition to the general requirements for data, the following are conditions that require reanalysis for a particular sample and analyte, beginning with the preparation or recounting, as appropriate:

7.4.1.1. If constant quench method of calibration is used, the quench of the sample shall be within  $\pm 5\%$  of the quench of the efficiency standard. If this condition is not met, the sample must be reanalyzed beginning with vial preparation.

7.4.1.2. If the sample quench does not fall within the range of the quench curve, the samples shall be re-analyzed such that the sample quench is in the range of a quench curve.

7.4.1.3. Site specific guidance may be provided for other methods such as counting “sample” and “sample plus spike”.

## **8. GAS FLOW PROPORTIONAL COUNTING**

### **8.1. Planchet Preparation**

8.1.1. Planchets shall be thoroughly cleaned before use to ensure that there are no interfering residues or contamination.

8.1.2. All planchets shall be prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves.

8.1.3. Sample weights shall be documented and stable prior to counting.

8.1.4. Planchets exhibiting physical characteristics notably different from the self-absorption standards (e.g., evidence of corrosion, solids on the sides or rim of planchet, etc.) shall not be counted unless remediation efforts such as additional sample preparation and remounting, flaming prove unsuccessful.

8.1.5. Any non-routine counting situations shall be documented in the Case Narrative.

### **8.2. Laboratory Control Sample (LCS)**

8.2.1. **Isotopes:** The isotopes used for the LCS for gross alpha/gross beta analysis shall be documented in the Case Narrative. Site specific guidance/requirements may be provided for isotopes to be used for the LCS.

8.2.2. **Evaluation Criteria:** For gross alpha/beta analysis, the acceptance criteria given in the *Quality Assurance/Quality Control section of this Attachment* is applicable when the analyte in the LCS is the same analyte used for the calibration curve. Site specific instructions will be provided when the LCS analyte is different from that used for the calibration curve.

### **8.3. Instrument Calibration**

8.3.1. **Instrument** calibration and usage shall be done in accordance with the requirements in ANSI N42.25, *Calibration and Usage of Alpha/Beta Proportional Counters*. Where the word “should” is used in ANSI N42.25, calibration shall be performed in accordance with the statement unless Site accepted justification is provided. References are for the customer determined current version. When references change, an implementation schedule will be determined.

#### **8.3.2. Calibration Sources and Standards**

8.3.2.1. The standard reference material(s) used to prepare sources for determining detector efficiencies and self-absorption curves shall be NIST traceable.

8.3.2.2. The source activities shall not be so great that the dead time of the electronics results in a large percentage(>5%) of lost events.

8.3.2.3. The geometry of the calibration sources used for efficiency and self-absorption/crosstalk curves shall be the same as that of the prepared sample and QC sample planchets. The depth and shape (flat, flanged, ringed, etc.), in addition to the diameter, are factors which shall be the same for calibration sources as for samples.

8.3.2.4. The sources used for the determination of self-absorption and cross talk should be of similar isotope content to that of the analytical samples. Site specific instructions may be provided regarding isotope content of samples and required isotopes to be used for calibration. If no direction is provided Am-241 or Th-230 shall be used for alpha and Cs-137 or Sr-90/Y-90 for beta.

### 8.3.3. **Instrument Backgrounds**

8.3.3.1. Instrument backgrounds for both alpha and beta shall be determined at least weekly.

### 8.3.4. **Self-Absorption and Cross-Talk Curves**

8.3.4.1. Self-absorption curves are required for both alpha and beta counting.

8.3.4.2. A cross-talk curve shall be established for alpha to beta cross-talk versus residue weight.

8.3.4.3. Beta to alpha cross-talk is not significantly affected by planchet residue weight, and is generally constant over the applicable weight range. Therefore this cross-talk correction does not require residue weight consideration.

8.3.4.4. The data used to generate self-absorption and cross-talk curves shall consist of at least 7 points, well distributed throughout the mass range.

8.3.4.5. Each alpha and beta calibration standard shall be counted to an accumulation of 10,000 counts.

### 8.3.5. **Check Source Requirements**

8.3.5.1. The alpha and beta calibration of each detector used to count analytical samples or QC samples shall be checked daily. The only exception to this requirement is when performing analyses with extended count times. In this case, check source measurements may be performed between sample sets.

8.3.5.2. Following gas bottle changes, check sources and backgrounds shall be analyzed and results shall be within control chart limits before samples are counted.

8.3.5.3. Check source data shall be evaluated per ANSI N42.25, documented and retained.

## 9. **TOTAL URANIUM BY LASER INDUCED KINETIC PHOSPHORESCENCE**

Site specific requirements will be provided when other methods for determination of total uranium are required.

### 9.1. **Selection Of Method**

9.1.1. **Sample Treatment:** Urine samples shall be at least evaporated to dryness and wet-ashed as described in ASTM Specification D 5174-91, *Trace Uranium by Pulsed-Laser*

*Phosphorimetry* prior to KPA measurement. Further requirements for additional clean-up of samples may be provided.

- 9.1.2. **Sample and Sample plus Spike Measurement:** For each sample, both the sample and sample plus spike shall be measured to demonstrate that there are no quenching interferences.

9.2. **Glassware And Water For Low Level Uranium**

- 9.2.1. For all low-level uranium analysis, prior to initial use, all new glassware with the exception of cuvettes used in KPA measurement, shall be soaked in hot 8 molar nitric acid for at least two hours and then in room temperature 8 molar nitric acid overnight. The upper limit for uranium concentration in the low level range will be Site specified.
- 9.2.2. ASTM Type II water shall be used to prepare but is not limited to standards, preparation of all reagents, and for final rinsing of glassware for items used in the determination of low level uranium.

9.3. **Preparation Requirements**

- 9.3.1. The sample preparation shall yield samples such that phosphorescence decay lifetimes fall in the range of 150  $\mu$ s to 350  $\mu$ s.

9.4. **Minimum Detectable Concentration (MDC) Determination**

The MDC is a function of the sample size, variability of the preparation blanks, and instrument background which is a combination of low-level signals produced by sources other than the analyte including: photomultiplier tube dark count (electronic noise), luminescence from the quartz cells and optics, impurities in reagents, and stray ambient light.

9.4.1. **MDC Calculation**

- The following equation shall be used to calculate the MDC:

$$\text{MDC} = [(4.65 * S_b) * \text{DIL}] / V$$

where:

- $S_b$  = standard deviation(1s) of the blank population
- $\text{DIL}$  = dilution factor (ratio of total sample taken for preparation/aliquot measured in the cuvette)
- $V$  = sample volume or weight (liters or grams)

Units are in  $\mu\text{g/l}$  or  $\mu\text{g/g}$

- The method of implementation of blank populations for calculation of MDCs shall be described in detail in an SOP which is accepted by the Site prior to use.
  - Note: Implementation of blank populations for KPA requires the use of Detection Limit Standards as the instrument is not capable of providing uranium concentrations for blanks. Specific procedures can be provided by the KPA vendor.
  - Site specific requirements for implementation of the MDC calculation may be provided.
- 9.4.2. The sample size used for blanks shall be the typical sample size for samples analyzed for this SOW. If a given sample size differs from the sample size used in the blanks, this

shall be addressed in the Case Narrative. It is not expected that sample size will differ from the sample size of the blanks.

- 9.4.3. The analyte MDC's as calculated above must be less than or equal to the RDL at the lowest dilution factor.

9.5. **Conditions Requiring Reanalysis**

- 9.5.1. **Sample and Analyte Specific Conditions:** the following are additional conditions that require reanalysis for a particular sample beginning with KPA measurement. If succeeding KPA measurement does not provide an adequate result, another aliquot shall be started with the sample preparation.

9.5.1.1. The lifetime of the phosphorescence is less than 150  $\mu$ s or greater than 350  $\mu$ s.

9.5.1.2. The linear regression coefficient of the decay plot is less than 0.96 for samples where the measured concentration is greater than the RDL. Every effort should be made to keep this greater than 0.98. It is expected that for most samples this will be greater than 0.99.

9.5.1.3. If the standard addition recovery is less than 90%, re-analysis is required:

$$\text{Standard Addition Recovery} = (\text{Conc. Spike} - \text{Conc. Sample}) + \frac{(\text{Std. Conc.} \times \text{Std. Vol.})}{\text{Sample Vol.} + \text{Std. Vol.}} \times 100$$

9.5.1.4. Analyte concentration is not in the range of the calibration curve used.

9.5.1.5. The Reference Ratio is less than 0.9 or greater than 1.1.

**Note:** The Reference Ratio is the ratio of laser intensity during sample measurement divided by its intensity during calibration. See KPA Manual for further explanations.

9.5.1.6. The continuing Calibration Check Standard is not within 10% of the known value.

9.6. **Instrument Calibration**

The purpose of the instrument calibration is to ensure that the KPA was initially capable of producing quality results according to the specifications given in this Attachment and that the calibration was maintained throughout the time period in which samples were analyzed.

9.6.1. The KPA shall be calibrated daily when in use.

9.6.2. At least three standards shall be used for each calibration range. The calibration range shall include the range of the samples to be measured.

9.6.3. The LCS shall be measured in the same calibration range as the samples in the batch. Observe the instrument manufacturer's recommendations for calibrating high and low ranges. If the measurements are performed in more than one calibration range, then a separate LCS shall be prepared for each range.

9.7. **Instrument Performance Check** (also referred to as the Calibration Check Standard)

The performance check using a standard prepared separately and a different concentration from the calibration standards, shall be performed upon completion of calibration and subsequently after every 10 samples are analyzed. The relative bias of the calibration check standard shall be in the range -0.10 to +0.10.

The performance check standard should be prepared from a different standard reference material than the calibration standards.

9.8. **Instrument Calibration Order**

The order of performing the Instrument Calibration shall be (1) Background (2) Calibration Curve (3) Calibration Check standard

9.9. **Calibration Requirements**

9.9.1. The instrument background shall be control charted with limits of acceptability and the background measurement shall fall within acceptable limits. Instrument background increases are not common. The more common problems are caused by dirty cell windows or contamination in the water or reagents.

9.9.2.  $R^2$  (linear regression coefficient) for the calibration curve shall be greater than or equal to .99.

9.9.3. The Calibration Check Standard shall be within 10% of the known value.

10. **GAMMA SPECTROMETRY**

10.1. **SAMPLE COUNTING REQUIREMENTS:** Procedures for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14-1991, *Calibration and use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides*, and/or ANSI N42.12-1994, *Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides*. References are for the customer determined current version. When references change, an implementation schedule will be determined.

10.1.1. **Detector Type:** The gamma detector system shall consist of any detector suitable for measuring the gamma isotopes of interest in the range of 0.06 to 2 MeV with regard to attaining RDLs, bias and precision requirements. Specific guidance/requirements may be provided on a case-by-case basis. Ge detectors of either intrinsic (pure) germanium or lithium drifted germanium are preferred; however for some specific requirements, another detector type, such as sodium iodide, may be more appropriate.

10.1.2. **Counting Geometry:** Detectors shall be calibrated for the specific geometry and matrix considerations used in the sample analysis.

10.1.3. **Spectral Acquisition, Processing and QC Software:** The Laboratory shall identify the software package(s) and versions used to analyze Site samples in the Case Narrative. If the Laboratory uses commercially provided software unmodified to process spectra and calculate gamma spectroscopy MDAs, documentation shall be provided upon request. If the Laboratory has modified the commercial software, or uses in-house developed software, a description of the software or modifications shall be Site accepted. The description shall include the algorithms and equations used for peak detection and fitting, nuclide identification, interference correction, energy and efficiency determination, and result, uncertainty and MDA calculation.

10.1.4. **Spectral Data Reference:** Identification of the reference used for the half-life, abundance and peak energy of all nuclides shall be documented.

10.1.5. **Spectral Background:** The count time for the background shall be at least as long as the sample count time. Background spectra shall be collected at the frequency prescribed for

Instrument Calibration. A background shall also be collected after any counting chamber changes have been made, i.e. cleaning, liner replacement, or instrument modification.

10.2. **Sample Preparation Requirements**

Sample preparation shall follow the Site specific requirements.

10.3. **QC Samples- Batch Blank**

Site specific instructions will be provided for the batch blank.

10.4. **QC Samples- Laboratory Control Sample (LCS)**

10.4.1. The LCS shall be traceable to the National Institute of Standards and Technology (NIST) or shall be a working reference material as described in *ASTM C 1128* and may be used repeatedly for different analytical batches as long as it is appropriate for the matrix and geometry of the batch.

10.4.2. The analyte need not be the same as the sample analyte but shall fall in the approximate energy region of the spectrum as the analyte(s) i.e. low, mid-range, or high energy.

10.5. **Instrument Calibration**

10.5.1. Efficiency Calibration Requirements

10.5.1.1. Each gamma spectrometry system used for Site samples shall be efficiency calibrated for the sample geometry and Site accepted matrix with NIST traceable standards or prepared from NIST traceable sources.

10.5.1.2. **Germanium Detectors:** Refer to *ANSI N42.14* for guidance on isotope specific efficiency and efficiency as a function of energy calibrations.

10.5.1.3. **Sodium Iodide Detectors:** Refer to *ANSI N42.12*.

10.5.2. **Energy Calibration Requirements**

10.5.2.1. **Germanium Detectors:** Refer to ANSI N42.14, paragraph 5.3.

10.5.2.2. **Sodium Iodide Detectors:** Refer to ANSI N42.12, paragraph 4.3.2.

10.5.2.3. The energy calibration for each gamma spectrometry system shall be checked each day of use. For systems using sample changers and/or long count times that run more than a day, the energy calibration shall be checked before each Analytical Batch.

10.6. **Performance Testing**

10.6.1. **Germanium Detectors:** Refer to ANSI N42.14, paragraph 7.

10.6.2. **Sodium Iodide Detectors:** Refer to ANSI N42.12, paragraph 4.3.5.

10.7. **SPECTRUM ASSESSMENT:** Each sample and QC sample spectrum shall be assessed for acceptability of key peak width and shape, and interference due to superimposed peaks or other sources. Any major contributor to the spectrum that is an unidentified peak shall be discussed in the Case Narrative.

## REFERENCES

### 1. REFERENCES

- 1.1. Site Internal Dosimetry Basis Manual
- 1.2. DOE Implementation Guide G-10CFR 835/C1 - Rev. 1, Internal Dosimetry
- 1.3. ANSI N13.30, Performance Criteria for Radiobioassay
- 1.4. Bioassay Intercomparison Study, Oak Ridge National Laboratory, Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6045
- 1.5. DOELAP Radiobioassay In-Vitro Pilot Study, Idaho Field Office, 785 DOE Place, Idaho Falls, ID 803401-1562
- 1.6. 10 CFR 1008 Privacy Act and (Public Law 93-579)
- 1.7. STD.ASTM C1128 Standard Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials, Feb., 1998
- 1.8. U.S., NUCLEAR REGULATORY COMMISSION REGULATORY GUIDE 4.15  
Revision 1, February, 1979.
- 1.9. Secretariat PROCORAD; 6, quai de Caligny; 50100; Cherbourg, France

### 2. SYNTHETIC URINE

#### 2.1. Synthetic Urine Recipe

The following lists the constituents and required quantities for synthetic urine to be used for matrix blanks and matrix LCSs when real urine from unexposed individuals is not used. This formulation is the one from which samples were prepared for the second round robin bioassay intercomparison study in support of draft ANSI Standard N13.30 "Performance Criteria for Radiobioassay". For routine urine bioassay line item codes found in the Bioassay Analyte Tables, at least 1200 mls of this matrix is required for the batch blank and batch LCS.

<u>COMPONENT</u>	<u>g/Kg.</u>
1. Urea	16.0
2. NaCl	2.32
3. KCl	3.43
4. Creatinine	1.10
5. NaSO4(anhyd.)	4.31
6. Hippuric acid	0.63
7. NH4Cl	1.06
8. Citric acid	0.54
9. MgSO4(anhyd.)	0.46
10. NaH2PO4·H2O	2.73
11. CaCl2·2H2O	0.63
12. Oxalic acid	0.02
13. Lactic acid	0.094
14. Glucose	0.48
15. Na2SiO3·9H2O	0.71
16. Pepsin	0.029
17. Conc. Nitric acid (70%)	50.00



### 3. SYNTHETIC FECAL

#### 3.1. Synthetic Fecal Matrix

The following lists the constituents and required quantities for synthetic fecal samples to be used for matrix blanks and matrix LCSs. The recipe is, in general, based on the formulation of the Department of Energy, Idaho Field Office Laboratory Quality Branch DOELAP program. In order to be able to homogenize the sample, peanut butter and water were substituted for the peanut oil. At least 65 g. of this matrix is required for each batch blank and batch LCS.

<u>COMPONENT</u>	<u>g/Sample</u>
1. Calcium hydroxide	0.97
2. Ferric ammonium sulfate	0.04
3. Magnesium carbonate	0.61
4. Potassium carbonate	0.83
5. Ammonium dihydrogen phosphate	2.1
6. Sodium sulfate	0.37
7. Ammonium chloride	0.04
8. Zinc sulfide	0.01
9. Leucine	7.1
10. Lysine	5.1
11. Methionine	0.8
12. Threonine	2.0
13. Palmitic acid	3.0
14. Stearic acid	2.0
15. Oleic acid	1.0
16. Cellulose	4.0
17. Gelatin	5.0
18. Peanut butter	10
19. H2O	20

## DATA PACKAGE COMPONENTS

### 1. RESULTS ONLY DELIVERABLE

The Bioassay Results Only deliverable deviates from the deliverable requirements of Attachment I of the BOA Statement of Work.. For Bioassay, the Sample Summary Sheet(s) described in Attachment I are not applicable and shall be replaced with the following with one sample reported per page:

**Table L-1: Bioassay Results Only Deliverable**

Component Name	Description
<b>Bioassay Results Only deliverable</b> <i>(See Section 1 of Attachment I to the BOA SOW)</i>	The described Cover page, Chain of Custody, and Case Narrative will be included. The Summary Sheets are not applicable and are replaced with Sample Summary Sheets as described below:
<b>Sample Summary Sheets</b>	<ul style="list-style-type: none"> <li>• LAST NAME, FIRST NAME, AND MIDDLE INITIAL of person providing the sample (as provided by the Site). <b>Note:</b> Provide a blank field for this information if a name is not provided by the Site.</li> <li>• EMPLOYEE NUMBER or SOCIAL SECURITY NUMBER of person providing the sample (as provided by the Site)</li> <li>• REPORT NUMBER assigned by the Site</li> <li>• SAMPLE NUMBER assigned by the Site</li> <li>• LABORATORY SAMPLE NUMBER (assigned by laboratory)</li> <li>• DATE OF SAMPLE RECEIPT</li> <li>• SAMPLE DATE as provided by the Site</li> <li>• SAMPLE TIME as provided by the Site</li> <li>• BIOASSAY TYPE (Urine, feces, nasal, tissue)</li> <li>• BIOASSAY RADIONUCLIDE requested</li> <li>• DATE OF SAMPLE RESULTS</li> <li>• MEASURED VALUE in disintegrations per minute (dpm)/sample for each analyte requested (tritium - pCi/L and for gross alpha for nasal swabs – cpm/sample)</li> <li>• MEASUREMENT UNCERTAINTY (1 sigma) in the same units as the measured value</li> <li>• PERCENT SAMPLE RECOVERY FOR THE ADDED TRACER</li> <li>• SUBMITTED SAMPLE SIZE in volume (ml) for urine or mass (g) for fecal</li> <li>• ALIQUOT SIZE ANALYZED (=1 if the total sample was used)</li> <li>• MINIMUM DETECTABLE AMOUNT (MDA) in the same units as the measured value</li> <li>• DECISION LEVEL (Lc) in the same units as the measured value</li> <li>• NOTATION of the sample validation by the laboratory (“V” for valid or “I” for invalid followed by date and validator’s signature)</li> </ul> <p>COMMENTS regarding sample processing, sample condition or information provided by the Site that is relevant to the bioassay result. The Sample Data Package Narrative may reference this comment.</p>

## 2. STANDARD DELIVERABLE

The Bioassay Standard Deliverable deviates from the deliverable requirements of Attachment I of the BOA Statement of Work. The following table describes the Bioassay Standard Deliverables:

**Table L-2: Bioassay Standard Deliverable**

<b>Component Name</b>	<b>Description</b>
<b>Bioassay Results Only Deliverable</b>	All components contained in the Bioassay Results Only Deliverable data package.
<b>Sample and QC Results Summary</b>	<p>The Sample and QC Summary shall be submitted for each batch including the required data for both the samples and the QC samples. It shall be submitted as one section per batch.</p> <ul style="list-style-type: none"> <li>• REPORT IDENTIFICATION NUMBER (RIN)</li> <li>• LAST NAME, FIRST NAME, AND MIDDLE INITIAL of person providing the sample (as provided by the Site)</li> <li>• EMPLOYEE NUMBER or SOCIAL SECURITY NUMBER of person providing the sample (as provided by the Site). This field will be NA for the batch blank and batch LCS and other applicable QC.</li> <li>• LABORATORY ID</li> <li>• ANALYSIS/ANALYTE (e.g. Urine/Pu239/240)</li> <li>• RESULT AND ERROR AT ONE STANDARD DEVIATION</li> <li>• UNITS (Isotopics - dpm/sample, Bioassay Smears - cpm/sample, Tritium in Urine – pCi/l)</li> <li>• YIELD</li> <li>• MDA</li> <li>• DECISION LEVEL (<math>L_c</math>)</li> </ul>
<b>QC Summary</b>	<p>The QC Summary shall contain the following information in a tabulated format:</p> <ul style="list-style-type: none"> <li>• LCS SAMPLE NUMBER</li> <li>• DATE OF ANALYSIS</li> <li>• KNOWN VALUE OF LABORATORY CONTROL SAMPLE (LCS)</li> <li>• OBSERVED LCS VALUE</li> <li>• RELATIVE BIAS</li> <li>• AVERAGE RELATIVE BIAS - List all LCS, their date of analysis, individual relative bias and calculate per Site Specific Instructions).</li> <li>• PRECISION - Calculated per Site Specific Instructions.</li> </ul>
<b>Blank Population Summary</b> (If Applicable):	<ul style="list-style-type: none"> <li>• Blank Sample Number</li> <li>• Date Of Analysis</li> <li>• Dpm Of Tracer Used In The Blank</li> <li>• Blank Result in cpm</li> <li>• Tracer Recovery</li> <li>• Detector Efficiency</li> <li>• Blank Result in dpm</li> <li>• Standard Deviation Of Blank Population (In Dpm)</li> <li>• Standard Deviation Of Blank Population (In Cpm)</li> <li>• Mean Blank Value Of The Blank Population (In Dpm)</li> </ul>

### 3. STANDARD PLUS RAW DATA DELIVERABLE

The following is a listing of hard copy deliverables required for the Bioassay Standard Plus Raw Data Deliverable category. This category contains all the components required for the Bioassay *Standard Deliverable* plus standards data, calibration data, preparation data and instrument raw data.

**Table L-3: Bioassay Standard Plus Raw Data Deliverable**

Component Name	Description
<b>Bioassay Standard Deliverable</b>	All components contained in the Bioassay Standard Deliverable data package
<b>Raw Data</b>	<p><b>Preparation Raw Data</b></p> <p>Sample preparation raw data shall be documented in the form of bench sheets and/or preparation logs containing, at a minimum, the following:</p> <ul style="list-style-type: none"> <li>• Analytical Batch identifier</li> <li>• Date of preparation</li> <li>• Identifier for the laboratory SOP for the preparation</li> <li>• Identifiers for all sample and QC samples in the batch</li> <li>• Identifiers that provide for traceability of tracer, LCS, matrix spike, etc. dilutions used</li> <li>• Concentration of standards used for tracer, LCS, matrix spike, etc.</li> <li>• Volumes or weights of added tracers, LCS analyte(s), matrix spike(s), etc.</li> <li>• Balance identifiers with dates of use</li> <li>• Initial and final weights and volumes for all samples and QC samples including gross weights, tare weights, and aliquot weights where applicable</li> <li>• Pipette identifiers and dates of use (if applicable)</li> <li>• Comments describing any significant sample changes or reactions which occur during preparation</li> <li>• Signatures and dates of all analysts and reviewers</li> </ul> <p><b>Standards Summary</b></p> <p>This section shall contain information for all standards used for data reported in the RIN. This shall include but is not limited to the tracer, analyte(s) in the LCS, matrix spikes, and the Instrument Calibration Standards used for efficiency and/or check sources.</p> <ul style="list-style-type: none"> <li>• Standard I. D. traced back to the primary standard I. D. (All identifiers must be traceable to standard reference material certificates. Submit only the first page of the NIST certificate to establish primary standard I. D. and/or traceability.</li> <li>• Standard isotope, concentration, and error</li> <li>• Expiration Date</li> <li>• Use for this standard(tracer, LCS, efficiency, etc.)</li> <li>• Date of preparation</li> <li>• Sufficient dilution data to provide for calculation of the activity</li> </ul>

**Table L-3: Bioassay Standard Plus Raw Data Deliverable (continued)**

Component Name	Description
	<p><b>Calibration Raw Data</b></p> <p>All associated raw data used to calibrate the instrument and for check sources for the period in which the samples were counted</p> <ul style="list-style-type: none"> <li>• Instrument Calibration SOP</li> <li>• Identification of Software used to produce Instrument Calibration</li> <li>• Data File Name for this Calibration</li> <li>• Energy Calibration data <ul style="list-style-type: none"> <li><b>Alpha Spec and Gamma Spec:</b> Energy Calibration date and isotopes used, calibration equation</li> <li><b>Liquid Scintillation Counting and KPA:</b> Not Applicable</li> <li><b>Gas Proportional Counting:</b> Date of voltage plateau and discriminator window settings and isotopes used</li> </ul> </li> <li>• Background Determination <ul style="list-style-type: none"> <li>➢ Date of Background</li> <li>➢ Length of background count</li> <li>➢ Background Counts</li> <li>➢ List ROI for each isotope of interest with counts in ROI (For Alpha Spec and Gamma Spec)</li> </ul> </li> <li>• Efficiency Determination <ul style="list-style-type: none"> <li><b>Alpha Spec:</b> Date of Efficiency Curve and isotopes used and efficiency</li> <li><b>Gamma Spec:</b> Date of Efficiency, isotopes used, and efficiency equation</li> <li><b>Liquid Scintillation Counting:</b> Date of quench curve or date of Efficiency calibration for constant quench, equation relating efficiency to Quench parameters. Include the daily measurements with appropriate control charts for Instrument Performance Assessment taken on count date of samples in this package.</li> <li><b>Gas Proportional Counting:</b> Date of efficiency/self-absorption Curve(s) and isotopes used</li> <li><b>KPA:</b> Date of Calibration Curve and Calibration Check Standard</li> </ul> </li> <li>• List of detector IDs calibrated on the above dates and with the above characterization. Do not report detectors which did not meet calibration limits of acceptability with regard to energy, background or efficiency.</li> </ul> <p><b>Sample Analysis Raw Data</b></p> <p>All raw data associated with the generation of sample results. This includes data for analyses performed but not used for reporting. It also includes raw data for matrix spike, duplicate, blanks, LCS and every sample in the batch. It shall include but is not limited to the following:</p> <ul style="list-style-type: none"> <li>• Sample ID (Site or Laboratory)</li> <li>• Date and time of analysis</li> <li>• Count Time</li> <li>• Data File Name</li> <li>• Instrument and detector ID</li> <li>• File Name of Background used</li> <li>• Appropriate detector background</li> <li>• Detector efficiency for this sample</li> <li>• Analytical Batch ID</li> <li>• Sample Aliquot Size</li> <li>• Analyte Isotope(s)</li> </ul>

**Table L-3: Bioassay Standard Plus Raw Data Deliverable (continued)**

<b>Component Name</b>	<b>Description</b>
	<ul style="list-style-type: none"><li>• Start and End channels for all applicable ROIs</li><li>• Analyte(s) gross counts</li><li>• Background counts (identify count time of background)</li><li>• Analyte(s) net counts</li><li>• FWHM and peak energy where applicable</li><li>• Where applicable list data also for tracer isotope described in last 6 bullets</li><li>• Channel by Channel spectral print-out for alpha spec that includes each ROI(including tracer) and an equivalent number of channels above and below the ROI</li><li>• Quench Indicating Parameter for Liquid Scintillation Counting</li><li>• Required KPA Raw Data will be defined by Site Specific Requirements</li><li>• Calibration Isotopes for gross alpha/beta</li><li>• Instrument Run Log for applicable count dates</li></ul>

**ATTACHMENT K (ADDENDUM)**

**TO**

**BASIC ORDERING AGREEMENT**

**ATTACHMENT 1**

**STATEMENT OF WORK**

**ITEM CODES**

# 1. GENERAL BIOASSAY ANALYTE LIST AND LINE ITEM CODES

The following list of bioassay line item codes will be incorporated into Attachment K to the Basic Ordering Agreement (BOA) Attachment 1, "Statement of Work". This list currently includes the Rocky Flats Environmental Technology Site (RFETS) General Bioassay Line Item Codes at this time.

Please use the attached General Bioassay Analyte Lists Form to add other analytes to this list.

Line Item Code	Title	Method	Matrix	Analyte(s)	CAS. No	RDL	Units
BAS-A-001	Plutonium 30 day	Alpha Spec	Urine	<sup>239,240</sup> Pu	10-12-81	0.02	dpm/sample
BAS-A-002	Plutonium 21 day	Alpha Spec	Urine	<sup>239,240</sup> Pu	10-12-81	0.06	dpm/sample
BAS-A-003	Plutonium 14 day	Alpha Spec	Urine	<sup>239,240</sup> Pu	10-12-81	.13	dpm/sample
BAS-A-004	Uranium 30 day	Alpha Spec	Urine	<sup>233, 234</sup> U	11-08-5	0.1	dpm/sample
				<sup>235</sup> U	15117-96-1		
				<sup>238</sup> U	7440-61-1		
BAS-A-005	Uranium 21 day	Alpha Spec	Urine	<sup>233, 234</sup> U	11-08-5	0.13	dpm/sample
				<sup>235</sup> U	15117-96-1		
				<sup>238</sup> U	7440-61-1		
BAS-A-006	Uranium 14 day	Alpha Spec	Urine	<sup>233, 234</sup> U	11-08-5	0.26	dpm/sample
				<sup>235</sup> U	15117-96-1		
				<sup>238</sup> U	7440-61-1		
BAS-A-007	Americium 21 day	Alpha Spec	Urine	<sup>241</sup> Am	14596-10-2	.06	dpm/sample
BAS-A-008	Americium 14 day	Alpha Spec	Urine	<sup>241</sup> Am	14596-10-2	0.13	dpm/sample
BAS-A-009	Plutonium 21 day	Alpha Spec	Fecal	<sup>239,240</sup> Pu	10-12-81	0.20	dpm/sample
BAS-A-010	Plutonium 14 day	Alpha Spec	Fecal	<sup>239,240</sup> Pu	10-12-81	1.30	dpm/sample
BAS-A-011	Americium 21 day	Alpha Spec	Fecal	<sup>241</sup> Am	14596-10-2	0.80	dpm/sample
BAS-A-012	Americium 14 day	Alpha Spec	Fecal	<sup>241</sup> Am	14596-10-2	5.20	dpm/sample
BAS-A-013	Plutonium Uranium 30 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.020	dpm/sample
				<sup>233, 234</sup> U	11-08-5	0.10	
				<sup>235</sup> U	15117-96-1	0.10	
				<sup>238</sup> U	7440-61-1	0.10	
BAS-A-014	Plutonium Uranium 21 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.06	dpm/sample
				<sup>233, 234</sup> U	11-08-5	0.13	
				<sup>235</sup> U	15117-96-1	0.13	
				<sup>238</sup> U	7440-61-1	0.13	



Line Item Code	Title	Method	Matrix	Analyte(s)	CAS. No	RDL	Units
BAS-A-015	Plutonium Uranium 14 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.13	dpm/sample
				<sup>233, 234</sup> U	11-08-5	0.26	
				<sup>235</sup> U	15117-96-1	0.26	
				<sup>238</sup> U	7440-61-1	0.26	
BAS-A-016	Plutonium Americium 30 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.02	dpm/sample
				<sup>241</sup> Am	14596-10-2	0.04	
BAS-A-017	Plutonium Americium 21 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.06	dpm/sample
				<sup>241</sup> Am	14596-10-2	0.06	
BAS-A-018	Plutonium Americium 14 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.13	dpm/sample
				<sup>241</sup> Am	14596-10-2	0.13	
BAS-A-019	Plutonium Americium 7 day	Alpha Spec	Urine	<sup>239, 240</sup> Pu	10-12-8	0.26	dpm/sample
				<sup>241</sup> Am	14596-10-2	0.26	
BAS-A-020	Plutonium Americium 2 day	Screen for Activity	Urine	<sup>239, 240</sup> Pu	10-12-8	100	dpm/sample
				<sup>241</sup> Am	14596-10-2	100	
BAS-A-021	Plutonium Americium 21 day	Alpha Spec	Fecal	<sup>239, 240</sup> Pu	10-12-8	0.2	dpm/sample
				<sup>241</sup> Am	14596-10-2	0.8	
BAS-A-022	Plutonium Americium 14 day	Alpha Spec	Fecal	<sup>239, 240</sup> Pu	10-12-8	1.3	dpm/sample
				<sup>241</sup> Am	14596-10-2	5.2	
BAS-A-023	Plutonium Americium 7 day	Alpha Spec	Fecal	<sup>239, 240</sup> Pu	10-12-8	2.6	dpm/sample
				<sup>241</sup> Am	14596-10-2	10.4	
BAS-A-024	Plutonium Americium 2 day	Alpha Spec	Fecal	<sup>239, 240</sup> Pu	10-12-8	100	dpm/sample
				<sup>241</sup> Am	14596-10-2	100	
BAS-A-025	Plutonium Americium 14 day	Alpha Spec	Tissue	<sup>239, 240</sup> Pu	10-12-8	1.0	dpm/sample
				<sup>241</sup> Am	14596-10-2	1.0	
BAS-A-026	Plutonium, Americium 7 day	Alpha Spec	Tissue	<sup>239, 240</sup> Pu	10-12-8	1.0	dpm/sample
				<sup>241</sup> Am	14596-10-2	1.0	
BAS-A-027	Tritium 30 day	Liquid Scintillation	Urine	<sup>3</sup> H	10028-17-8	600	pCi/l
BAS-A-028	Tritium 21 day	Liquid Scintillation	Urine	<sup>3</sup> H	10028-17-8	600	pCi/l
BAS-A-029	Tritium 14 day	Liquid Scintillation	Urine	<sup>3</sup> H	10028-17-8	600	pCi/l
BAS-A-030	Gross Alpha Screen 5 day	Liquid Scintillation	Nasal/Mouth Swabs	Gross Alpha	12587-46-1	30	cpm
BAS-A-031	Gross Alpha Screen 3 day	Liquid Scintillation	Nasal/Mouth Swabs	Gross Alpha	12587-46-1	30	cpm

Line Item Code	Title	Method	Matrix	Analyte(s)	CAS. No	RDL	Units
BAS-A-032	Gross Alpha Screen 1 day	Liquid Scintillation	Nasal/Mouth Swabs	Gross Alpha	12587-46-1	30	cpm
BAS-A-033	Plutonium Uranium Americium 21 day	Alpha Spec	Nasal/Mouth Swabs	<sup>239, 240</sup> Pu	10-12-8	1	dpm/sample
				<sup>233, 234</sup> U	11-08-5	1	
				<sup>235</sup> U	15117-96-1	1	
				<sup>238</sup> U	7440-61-1	1	
				<sup>241</sup> Am	14596-10-2	1	
BAS-A-034	Alpha Spec Recount	Alpha Spec	Various(see original matrix)	Various(see original Analyte)	Various (se original CAS NO.)	Variable(see original RDL)	Variable(Se e original units)
BAS-A-035	Liquid Scintillation Recount	Liquid Scintillation	Various(see original matrix)	Various(see original Analyte)	Various (se original CAS NO.)	Variable(see original RDL)	Variable(Se e original units)
BAS-A-036	Gas Flow Proportional Recount	Proportional Counter	Various(see original matrix)	Various(see original Analyte)	Various (se original CAS NO.)	Variable(see original RDL)	Variable(Se e original units)
BAS-A-037	Gamma Spec Recount	Gamma	Various(see original matrix)	Various(see original Analyte)	Various (se original CAS NO.)	Variable(see original RDL)	Variable(Se e original units)